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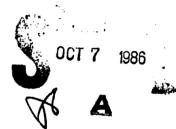
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TECHNICAL REPORT BRL-TR-2754

STUDIES OF POWDERED METAL SINTERING AND APPLICATIONS TO COMBUSTION DIAGNOSTICS

Mark A. DeWilde



September 1986

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REPORT DOCUMENTATION PAGE	READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER 2. GOVT ACCESSION NO.	3 RECIPIENT'S CATALOG NUMBER
Technical Report BRL-TR-2754 AD-A17249	2
4. TITLE (and Subtitle)	TYPE OF REPORT & PERIOD COVERED
Studies of Powdered Metal Sintering and Applications to Combustion Diagnostics	Final
Applications to compastion biagnostics	6. PERFORMING ORG. REPORT NUMBER
	S. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(a)	8. CONTRACT OR GRANT NUMBER(4)
Mark A. DeWilde	
9. PERFORMING ORGANIZATION NAME AND ADDRESS	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
US Army Ballistic Research Laboratory	AREA & WORK UNIT NUMBERS
ATTN: SLCBR-IB	1L161102AH43
Aberdeen Proving Ground, MD 21005	
11. CONTROLLING OFFICE NAME AND ADDRESS	12. REPORT DATE
U.S. Army Ballistic Research Laboratory ATTN: SLCBR-DD-T	September 1986
Aberdeen Proving Ground, MD 21005-5066	13. NUMBER OF PAGES
14. MONITORING AGENCY NAME & ADDRESS(If different from Controlling Office)	15. SECURITY CLASS. (of this report)
	Unclassified
	154. DECLASSIFICATION DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report)	
Approved for Public Release; Distribution Unlim	ited.
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different fro	m Report)
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18. SUPPLEMENTARY NOTES	2,10
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19. KEY WORDS (Continue on reverse side if necessary and identify by block number,	
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Hemi-head Burner 5,	
Sintering	-
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)	meg. jmk
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The sintering process for Bronze materials is describe fabrication of flat flame burners, besides that	ibed. Its application to
the fabrication of flat flame burners, hemi-head bur detailed. Conclusions regarding porosity, mechanica	rners and flow meters are
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ACKNOWLEDGEMENTS

The author wishes to thank Mr. Steven Bunte for providing the profile shown in Figure 16. In addition, thanks go to the BRL machine shop for the tedious and dirty job of fabricating the numerous burner parts, and sintering molds. Finally, I wish to thank Ms. Mary Griffith and Mrs. Janet Krokowski our typists, for patiently reformatting and preparing this report for publication.

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I. INTRODUCTION

Powder metallurgy is an old art that has found uses in a wide range of areas from manufacture of filter elements, to the cheap production of parts for handguns. It is considered an art since not all properties of the produced material can be predicted in advance given a certain set of starting parameters. With this in mind, a study was undertaken to find a set of working parameters that would yield reproducible and predictable porosity in the finished product. The project was aimed at solving some special needs that studies in combustion diagnostics brought about. The results of the studies yielded three useful instruments that will be discussed in detail in the following sections: The flat flame burner, the hemi-head burner and the porous metal plug flowmeter/filter/damper.

II. MATERIALS, SUPPLIERS, AND SPECIAL FACILITIES

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The materials used for sintering metal were all of the bronze family. For the two burners described, powdered bronze from Alcan Ingots and Powders under the name "MD-68 HP filter powder" was used. This material is in the 60 to 80 mesh size range (0.030 to 0.177 mm) and is roughly spherical in shape. For the porous plug flowmeters, powdered bronze mixed fines (80 mesh and smaller) from Connecticut Engineering Associates was used, 80% copper, 20% tin. This powder was sieved into various mesh sizes, and will be referred to in the standard nomenclature of the powder metallurgist. Under this scheme, mesh sizes are preceded by a "+" or a "-" indicating that the powder is too large to pass through the holes of the indicated mesh in the former case, or will pass through in the latter. As an example, the nomenclature "-80 to +100" indicates that the powder will pass through an 80 mesh (per inch) screen, but not through a 100 mesh screen.

The material that all molds were constructed of was a manufactured graphite from the Poco Division of Union Carbide, grade DFP-1. This material is freely machinable and can be held to the closest of tolerances. The advantages of graphite over other possible mold materials include low cost, ease of fabrication, low toxicity and good dimensional stability over wide temperature ranges. The primary disadvantages were fragility (although not overly so) and the messiness associated with machining and handling graphite.

Two special purpose instruments to carry on this work were made: an "air conditioned" glove box, and a small laboratory reduction furnace. The glove box was simply an air-tight aluminum box with a plexiglass front plate through which standard gloves passed in order to manipulate objects within the box. Within the box was placed a standard laboratory muffle furnace capable of 1000°C for heating small parts to be shrink-fitted together. Since this heated the box intolerably, a tank of liquid nitrogen connected through a solenoid valve controlled by a thermostat into the chamber was used to both maintain a reasonable temperature and provide an inert atmosphere. In addition, asbestos gloves were worn to protect the operator's rubber-gloved hands from excessive radiative heating. The hydrogen reduction furnace used in this work was a converted lab muffle furnace. An insert was made that slid inside the muffle, and contained a flowing atmosphere of hydrogen. This atmosphere was heated in the muffle, and allowed the sintering of materials

placed in the interior in a reducing atmosphere. Figure 1 shows an illustration of the unit, and the following parts are called out:

- 1. Commercial muffle furnace
- Welded stainless steel insert with water cooled front flange

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- 3. Pressure measurement, pumpout and gas burn-off pipe
- 4. Hydrogen supply line
- 5. Vacuum pump
- 6. Vacuum valve
- 7. Burn-off port valve
- 8. Vacuum/pressure gauge
- 9. Porous metal gas burn-off plug
- 10. Porous plug water cooling line
- 11. Flowmeter, 0-1 LPM
- 12. Metering needle valve
- 13. Hydrogen shut-off valve
- 14. Tank regulator
- 15. Hydrogen supply tank
- 16. Rubber gasket
- 17. Bolt

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- 18. Faceplate
- 19. Firebrick
- 20. Water cooling line for faceplate

Operation of the furnace is simple. The objects to be heated are placed within the insert (2) (which permanently resides in the furnace (1)). faceplate (18) is bolted on with gasket (16) in place to the front flange of the insert. Water cooling is started on the front plate (18), the insert front flange (2) and the porous plug (9). The firebrick (19) is held to the faceplate (18) with Dow-Corning RTV silicone rubber compound and serves to reduce heat loss from the muffle and associated temperature nonuniformities. To start up, valves 13 and 7 are closed, and valves 6 and 12 are opened to evacuate the entire system. When the vacuum/pressure gauge (8) indicates less than 1 mm Hg. pressure, valve 6 is closed and valve 13 opened to admit hydrogen gas to the system. When gauge 8 reaches 1 atmosphere, valve 7 is opened, and the escaping hydrogen gas ignited so that it burns off as a harmless diffusion flame. The pore size in the plug is 15 microns, which is well below the quenching diameter for hydrogen gas so that there is no chance of a backfire into the chamber. Valve 12 is adjusted for a flow of 0.6 1pm. At this point, heating of the muffle is begun. Shut down after cooling is done by stopping the hydrogen flow, closing off the burn-off port valve, evacuating the chamber, backfilling with air through the burn-off port, and then removing the objects.

III. THE FLAT FLAME BURNER

One of the continuing problems in the study of combustion processes is the production of stable, reproducible flames for study. The primary difficulty is due to the wide range of flame speeds that are encountered in such work, and the narrow range of these speeds that most burners will stabilize. Another source of difficulties arises whenever a flame system containing one or more materials that are not in the gas phase at normal temperatures is to be studied, for instance the formaldehyde flame. This

Figure 1. The Hydrogen Reduction Furnace

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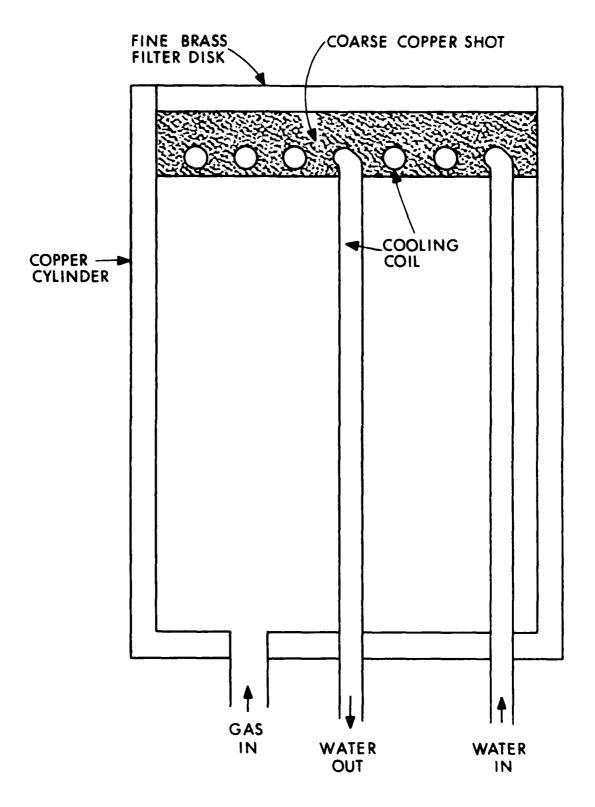
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material tends to polymerize to paraformaldehyde (a solid) and plug all burner open channels. To avoid this situation, all components in contact with the formaldehyde gas are kept at 100°C. This technique works well with other fuels such as methanol as well as other alcohols. Methanol flames are a rich source of formal radicals, and so are useful for studies of that propellant-related radical. As it turns out, one solution to the first of these two problems was solved in 1959 by Botha and Spaulding with their invention of the flat flame burner.

A typical design for a flat flame burner is shown in Figure 2. operation, the premixed fuel and oxidizer gases are introduced into the chamber below the sintered plug. The gases pass through the plug and are burned above it. To describe the mechanism of flame stabilization, we refer to the schematic model of the burner in Figure 3. Vo is the velocity of the gas mixture leaving the porous plug. VI is the velocity of the flame front spacing between the flame front and burner head, and O is the heat lost to the burner head from the flame front. The composition of the gas mixture, M, is assumed constant as is Vo. As the flame front moves toward the burner head, S decreases and Q increases. As O increases, the temperature, T, of the flame front decreases so that we may say that T is a function of S. As the flame temperature T decreases, the flame speed VI also decreases so that VI is also a function of S. The smaller S, the lower VI becomes. At some value of S, the flame speed VI will become equal to the exiting gas velocity Vo, and the flame front will cease to approach the burner head and become stable. As the feedback mechanism is determined by S, and the burner head is flat, the flame front is also flat.

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The argument for the creation of the flat flame requires that the exiting flame gas velocity Vo is everywhere constant, and that the temperature of those gases is also everywhere constant so that preheating effects do not alter local chemistry. In reality, various temperature and velocity gradients are found 2,3 and are caused by a number of situations. If no method is employed to reduce heat loss around the outer edge of the plug, or the plug is outer edge cooled as in old designs, that part of the plug is cooler than the inner parts. In addition, if some method is not provided to reduce the entrainment of room air in this same area, the flame will become more dilute in these portions and have a different flame velocity from the inner parts of The result of these two problems is an upturning of the flame edges so that a "dish"-shaped flame is actually produced. An additional source of error that Kaskan4 solved with his design stems from the fact that heat is applied to the burner surface everywhere in an even fashion, but removed in the original Botha and Spaulding design only at the edges. Kaskan solved this by embedding a cooling coil in the burner beneath the surface and attained a reduction in the temperature gradients found. The coils will, however, cause a certain amount of "shadowing" in the gas flow when measured at the burner surface. Ideally, the coils should be as close as possible to the surface for optimum heat removal (as sintered metal is a poorer conductor of heat than the bulk metal), but should be as far as possible away from the surface to minimize the effects of shadowing. It was found that if the coils were 7 diameters of the coil beneath the surface, shadowing was insignificant and cooling adequate for most flame systems. The problem of room air entrainment is minimized by providing an inert gas shroud around the burner, as is shown in Figure 4, along with the rest of the flat flame burner that resulted from this work.



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Figure 2. Kaskan's Rottom Cooled Flat Flame Burner

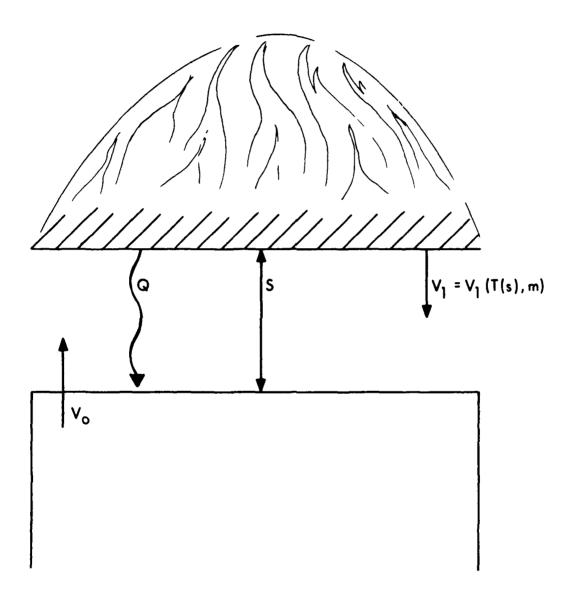


Figure 3. Flame Stabilization by Heat Extraction

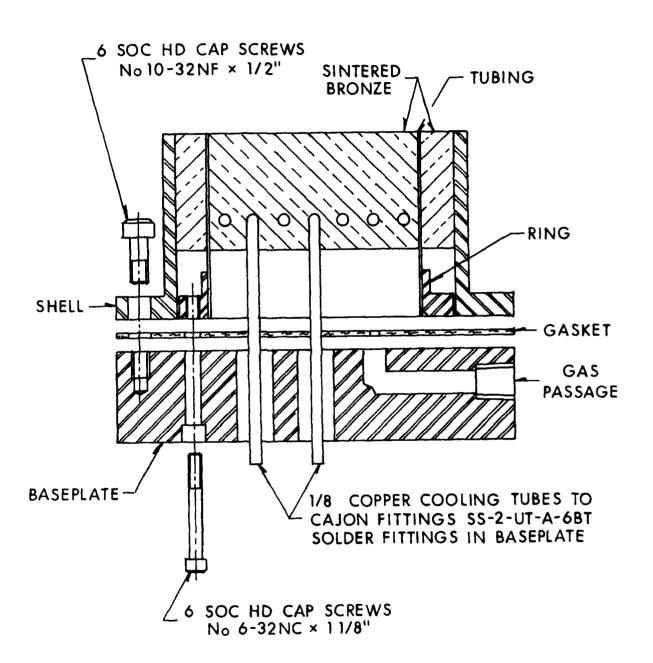


Figure 4. Composite Burner View

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The actual fabrication of the burner required that numerous samples of the bronze actually used be test-sintered, and the shrinkage, porosity, and mechanical strength be measured. Microscopic examination of these samples also provided useful information about the material. Sintered material that exhibits poor mechanical properties and tends to crumble like sandstone appears much the same as the metal powder before sintering. Local weak spot welds appear to be the marginal mechanism which holds the material together. As the sintering process produces better mechanical properties, examination indicates that the size of these spot welds increases, and the spheres become flattened at the weld points. Continuing the sintering process to yield high mechanical strength materials yields a sponge-like appearance under examination. A majority of the spheres have elongated and merged with their neighbors to form high strength bonds. It is to be noted that sintering is not a melting process, but rather an accelerated diffusion process. Too high a temperature and sintering time, and the plug becomes essentially a solid block of the material. Too little time at the correct temperature results in a properly porous sintered shell filled with a sandstone-like material. low a temperature for almost any time results in a material with sandstonelike properties. For these burners, it was found that sintering the Alcan "filter powder" at 840°C for four hours provided optimum porosity and mechanical strength. A composite view of the completed burner is shown in Figure 4. To make the center portion, first the spiral cooling coil is bent out of 1/8 inch 0.D. copper tubing with the aid of a jig. center sintering mold is shown in Figure 5. This is made of graphite and initially filled to a depth of 7/8 inch with the filter powder. Then the cooling coil is set in place, positioned with the sliding piston and the piston removed. Next, an additional 1/8 inch of filter powder is added, and the piston re-installed. Weights made of tungsten bar stock are placed on the piston, and the whole assembly placed in the reduction furnace and sintered. When cool, the center is of diameter 2.260 to 2.290 inches, and is ready to be placed within the inner chamber septum tube shown in Figure 6. In order to insure a gas-tight fit between the septum tube and burner center, the septum is shrink-fitted to the center. Due to the large amount of shrinkage required, the septum is heated to 1000°C. At this temperature, and due to the thinness of the walls of the tubing, the tube cools within seconds to a temperature too cool to allow successful shrink fitting. In addition, at these temperatures, the tube is easily deformed and made useless. circumvent both of these problems, a heat reservoir heat shrink method was devised. To the author's knowledge, this is the first use of this technique. A graphite heat reservoir, shown in Figure 7 is made. tube is placed inside, and the whole assembly placed in the furnace for heating. Before heating, there is appreciable space between the graphite and septum. Due to the large difference in thermal expansion coefficients between the graphite and stainless steel, after heating, the septum is tightly in contact with the heat reservoir which serves the three purposes of keeping the thin-wall tube hot, maintaining the septum's circular cross section, and providing a way to handle the septum without distorting it. In practice, the burner center is cooled in liquid nitrogen, and placed top surface down on a flat surface. Next, the reservoir-septum assembly is removed from the furnace at 1000° with tongs and quickly placed over the burner center. Within seconds, the rapid cooling of the septum by proximity with the cooled burner center causes it to shrink away from the reservoir, allowing the reservoir to be removed (still hot) from the septum. The septum then cools rapidly without appreciably heating the burner center, and the whole assembly is allowed to

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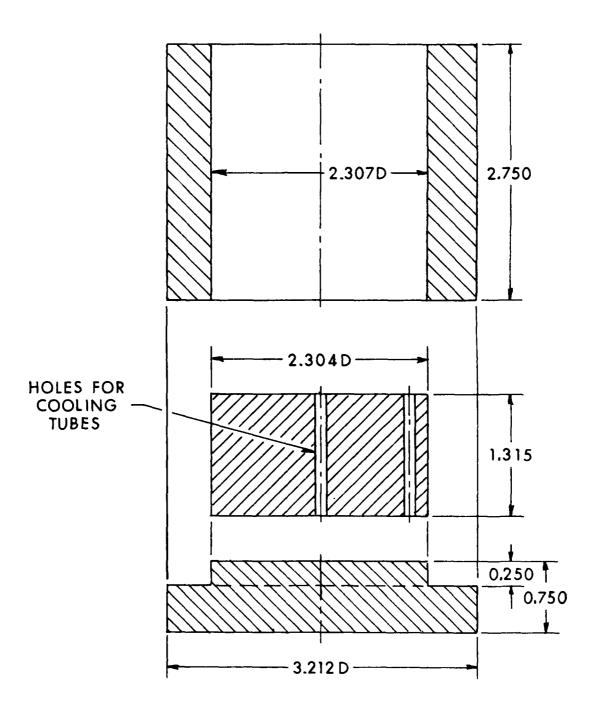


Figure 5. Burner Center Sintering Mould

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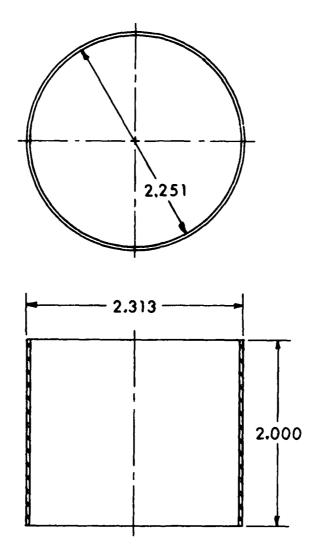


Figure 6. Inner Chamber Septum Tube

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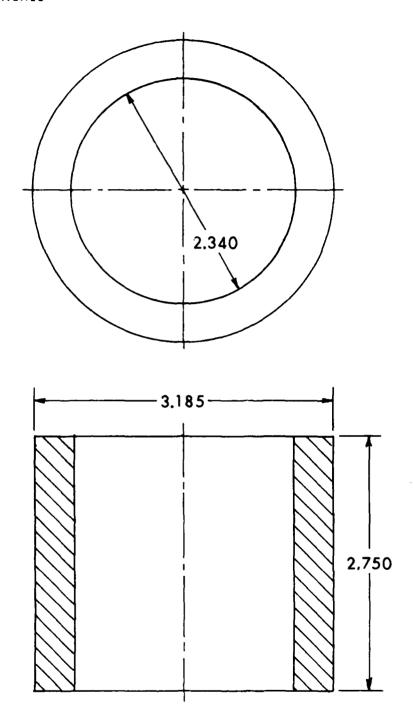


Figure 7. Graphite Heat Reservoir

warm to ambient temperature. Since the septum also expanded along its length when heated, as it cools it shrinks slightly in this direction, allowing the burner top surface to extend slightly above the septum when all assembly is complete. This is a highly desirable feature in that it prevents the septum from interfering with flame observations close to the burner surface.

The next stage in construction is the fabrication of the shroud ring. This component has requirements for greater strength than the burner center, and accordingly is sintered at a higher temperature for about the same period The ring was sintered for four hours at 900°C in the mold shown in Figure 8. The mold is assembled and filled to a depth of 3/4 inch with the filter powder. Next, the piston is placed in the annulus between the mold center piece and outer piece, and tungsten bar weights placed on top. whole assembly is then placed in the reduction furnace and sintered. cool, a hydraulic press is used to press the ring off the mold center piece. This ring is then placed in the glove box furnace and heated to 750°C. When hot, it is placed top side down on an insulating flat surface, and the burner center-septum assembly placed quickly in the center of the ring. This calls for some care, in that misalignment causes almost irrecoverable damage to the assembly. When the assembly is cool, its outermost diameter is measured and the inside diameter of the outer burner shell shown in Figure 9 is machined to that diameter less 0.010 inches. Next, the outer burner shell is placed in the glove box furnace, and heated to 1000°C to prepare for shrink fit. When hot, it is dropped into place over the top side down burner assembled to this point. Care must be taken in alignment both to prevent damage to parts, and to be sure that the cooling coil leads will go through the appropriate holes in the mounting plate (Figure 10).

The final stage of assembly is to align the inner sealing ring shown in Figure 11. on the inner septum tube so that the bolt holes are on radial lines with the bolt holes of the outer burner shell. When aligned, the inner sealing ring is silver-brazed to the inner septum tube in the chamfered The completed burner head is then bolted to the burner mounting plate shown in Figure 11, with the gasket shown in Figure 12 in place. Sealing of the 1/8 inch water cooling lines through the mounting plate is done by O-ring type vacuum fittings (cajon type SS-2-UT-A-6BT were used in this work). to be noted that all shrink fitting and heating of parts for shrink fitting were done in the nitrogen dry box as previously mentioned. This burner is capable of operating to temperatures of 300°C when teflon 0-rings and gaskets are used, making possible use with a wide variety of vaporizable fuels. When used for our work with the formaldehyde/nitrous oxide flame, a special oil recirculator cooling system was used. Briefly, this system pumped oil that was kept at a temperature of 110°C through the burner cooling coils. When the burner had warmed completely, the formaldehyde/N2O mixture was admitted to the burner chamber and ignited above the sintered metal plug for studies of that flame system.

IV. THE HEMI-HEAD BURNER

The flat flame burner mentioned above was used heavily for a large number of measurements taken in the primary and secondary flame zones of various gas mixtures. A difficulty arose, however, when it became desirable to probe the regions before the primary flame front in the unburned gas mixtures -- the so-

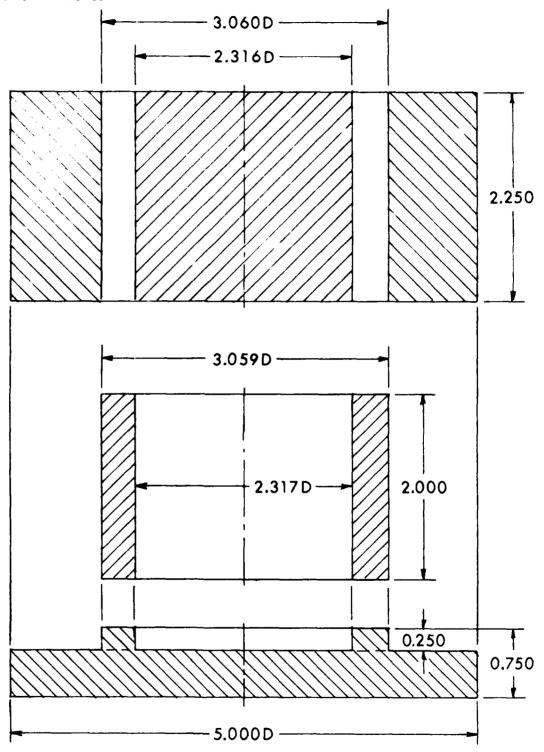


Figure 8. Shroud Ring Sintering Mold

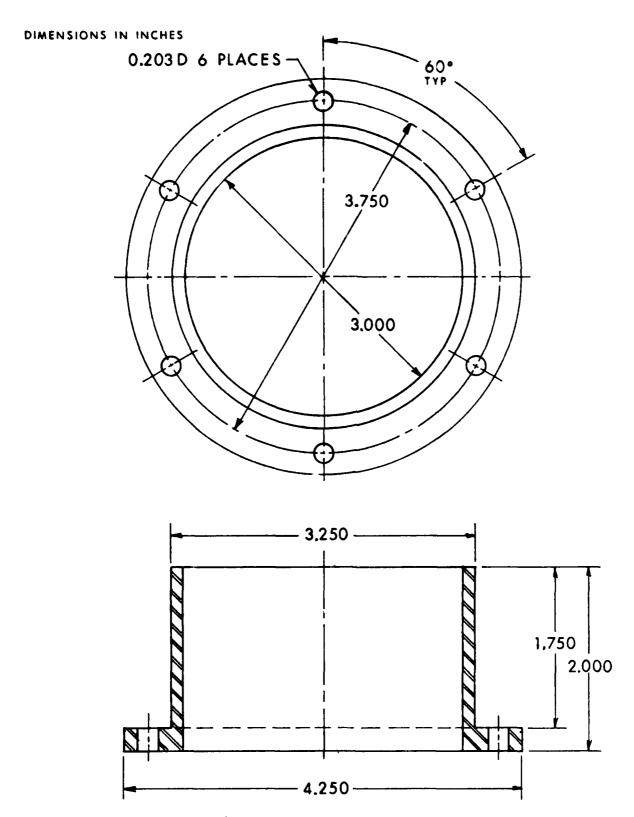


Figure 9. Outer Burner Shell

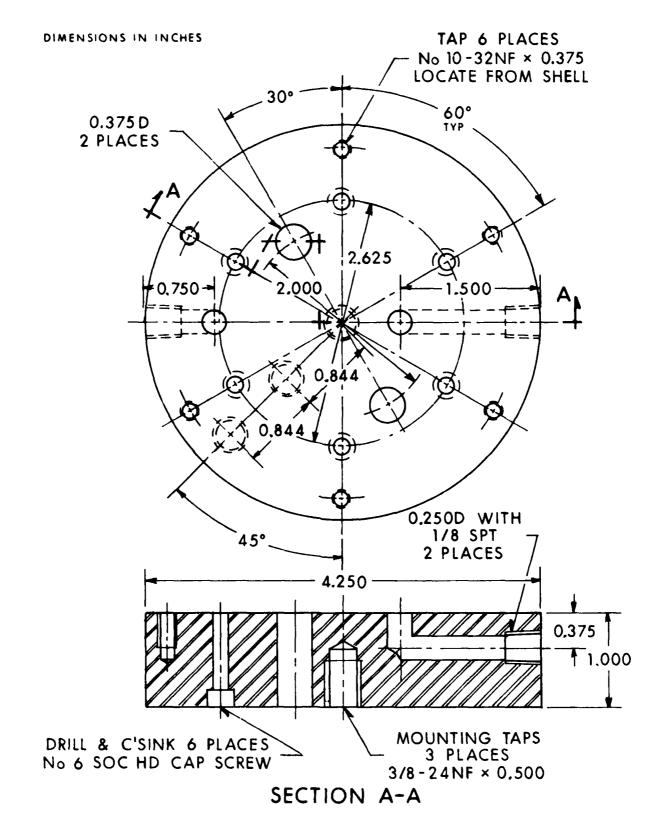


Figure 10. Burner Mounting Plate

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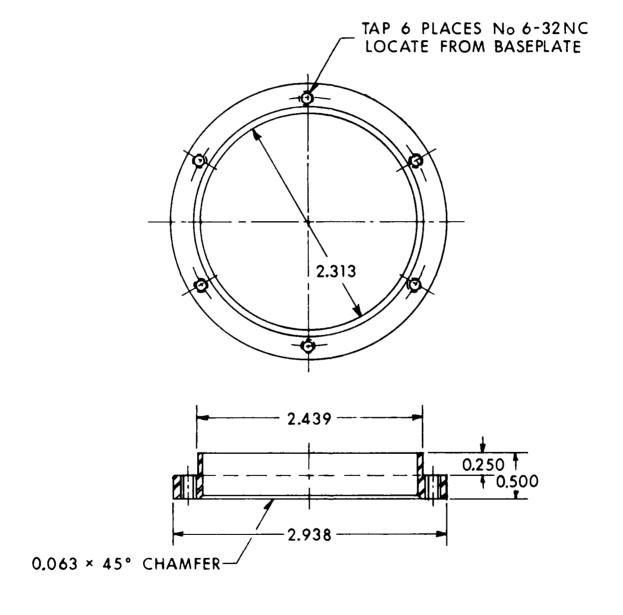


Figure 11. Inner Sealing Ring
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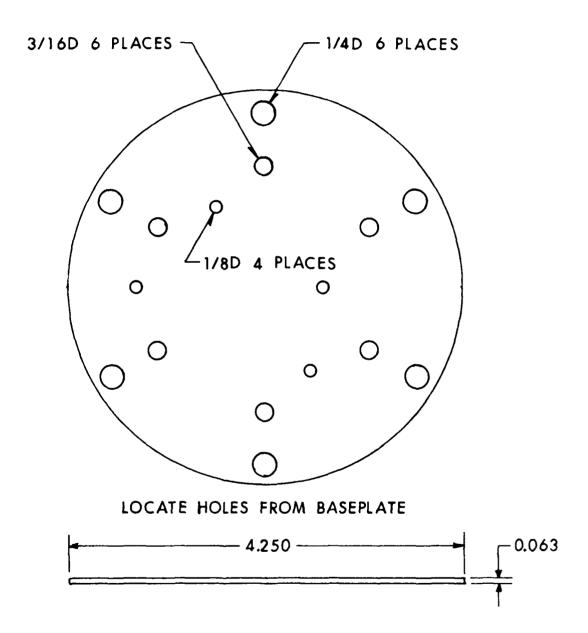


Figure 12. Burner Mounting Gasket

called preheat zone. Referring to Figures 13a and 13b, a laser probe beam enters from the left. Due to the change of refractive index at the flame boundary, the beam is deflected downwards and exits the flame at a lower level than it entered. If an attempt is made to probe lower in the flame as in 13h. the laser beam strikes the burner surface and the scattered light produced obscures all desired signal light produced by the probe (e.g., Raman spectra). This effectively means that smaller diameter burners must be used which are more susceptible to temperature non-uniformities and air entrainment, since the center of the burner where the probe is looking is isolated from the outside world by the surrounding flame. With all but the slowest flame speed gas mixtures, probing of early regions of the flame are impossible. It is only since the distance S of low flame speed gases is so large that these mixtures may be studied. One solution to the problem was to abandon the porous-plug burner altogether, and go to a new type of burner. Such an attempt was made, and resulted in the double knife-edge burner described elsewhere. The major disadvantage of this type of burner is the limited range of flame speeds that can be accommodated. The only way a fast flame such as hydrogen-air can be operated is by diluting and cooling the flame thus reducing the flame speed. As an alternative, a new modification to the old flat flame burner was developed: the hemi-head burner shown in Figure 14.

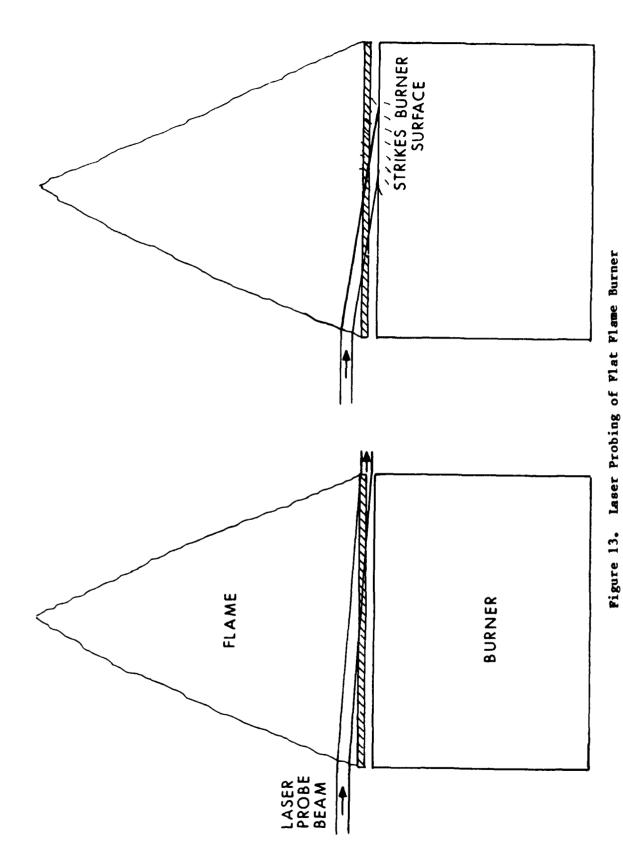
Fundamentally, the hemi-head burner is similar to the flat-flame burner in all respects. The burner parts with the exception of the burner center and burner center sintering mold are the same as for the flat flame burner. The sintering mold is shown in Figure 15. The 2-inch radius of curvature was chosen to match that same radius which was used in its double-knife edge counterpart. The assembly procedure needed alteration since it was no longer possible to use a flat surface as a reference plane. It is admitted that a flat insulating plate with a spherical depression to match the burner head could have been fabricated, but overall, since precision of assembly is relaxed for this burner, an alternative technique was used.

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After sintering the burner center and cooling to ambient, it is removed from the furnace, and cooled in liquid nitrogen to 77° Kelvin. The diameter is then measured, and the difference of that dimension and the burner inner septum tube computed. This quantity minus 0.002 inches is machined off the diameter of the burner center when warmed to ambient temperatures. Next, the center is again cooled in liquid nitrogen and measured. Additional cuts may be taken until the plug measures 0.002 inch larger than the inner septum inside diameter. Next, the inner septum tube is heated to 300°C and slid over the burner center into position and allowed to come to ambient. The remainder of the burner assembly follows the flat flame burner as described above. As proof of the ability to probe early flame regions, a spontaneous Raman spectra temperature profile of a methane-nitrous oxide flame is shown in Figure 16. The inflection and rapid rise of temperature near the burner head readily attests to the probing of early combustion processes.

V. THE SINTERED PLUG FLOWMETER

As any research burner requires, accurate flow measurements are necessary in order to characterize and reproduce any flame system. Previous work done along these lines resulted in the selection of capillary flowmeters. In



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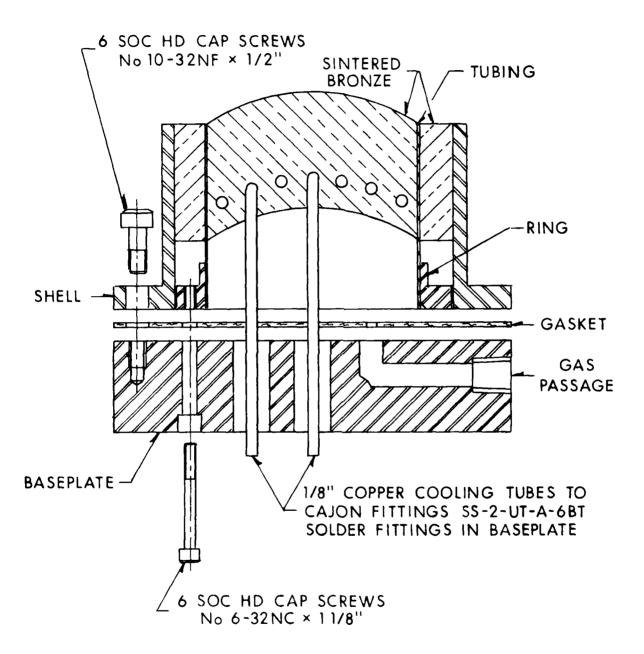


Figure 14. Hemi-Head Burner Cutaway View

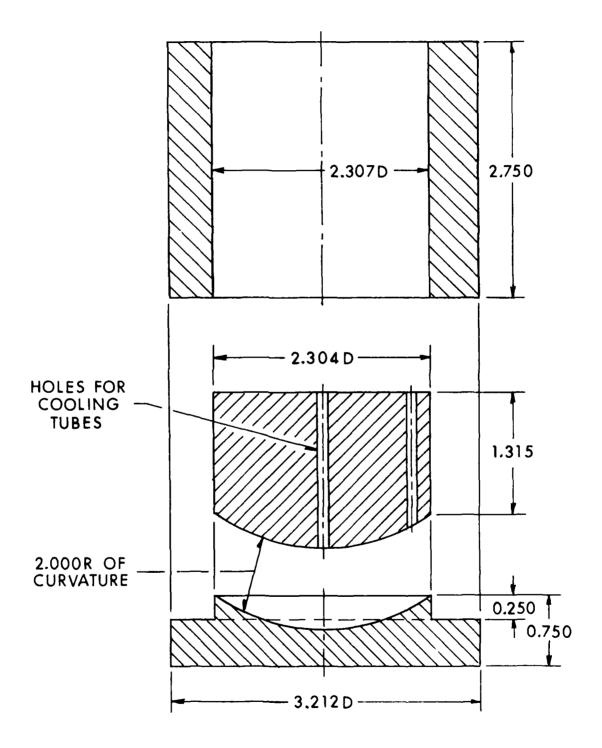
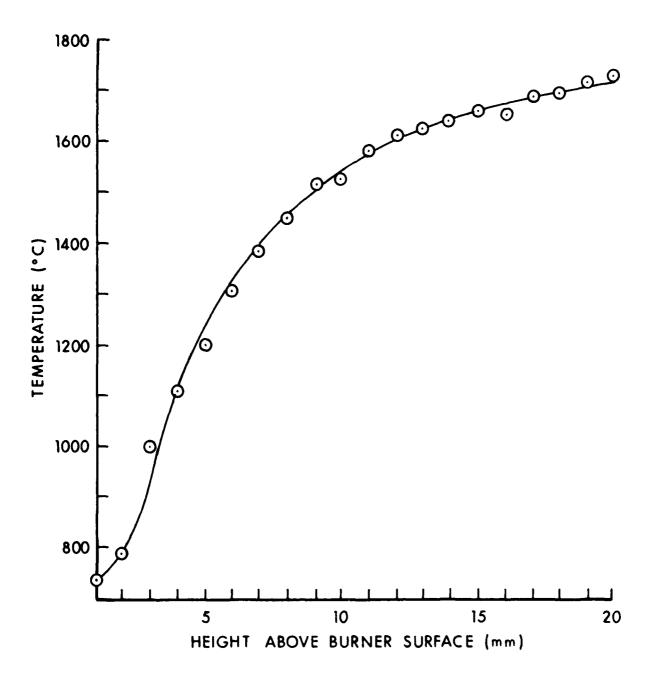


Figure 15. Hemi-Head Burner Center Sintering Mould



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Figure 16. Raman Temperature Profile of Methane Air Flame on Hemi-Head Burner 22

these devices, a small diameter capillary tube is used as a restriction, and the pressure drop across the capillary as a function of flow measured and fitted to a smooth curve. The primary drawback of such devices is a sensitivity to contamination. Even the smallest of particulate matter entering the capillary causes major changes in the flow vs. pressure drop calibration, and causes erratic operation. As an additional note, the calibration curves of these devices usually take the form of a second or third order polynomial and only rarely approximate linear. In order to solve the contamination problem, filters were sintered of the same material as the burners, and worked to reduce the problem to insignificant levels. The second problem was attacked by a study of the porosity of sintered metal plugs of various mesh sizes, and degrees of sintering. The reduction furnace being the same, only new molds and sintering material were needed.

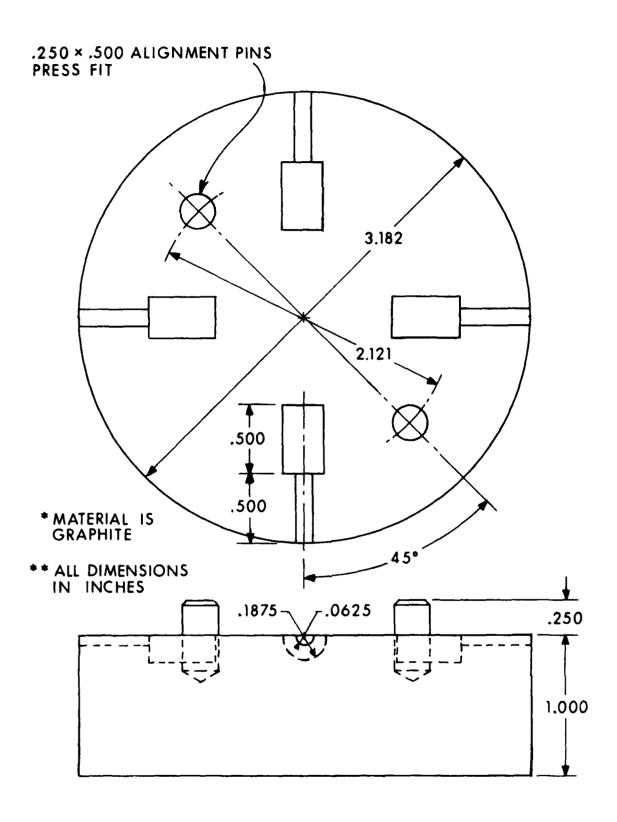
The sintering mold for the plugs is shown in Figures 17 and 18. The cylindrical cross section chosen was in all likelihood inferior to a spherical one, but machining difficulties forced the decision. The metal powder from Connecticut Engineering Associates was sieved to the following ranges of sizes:

Series	1	-	-100 to +140
Series	2	-	-140 to +170
Series	3	-	-170 to +200
Series	4	-	-200 to +325
Series	5	-	-325 to +400
Series	6		-400 to +500
Series	7	-	-500 to +625
Series	8	-	-625 mixed fines

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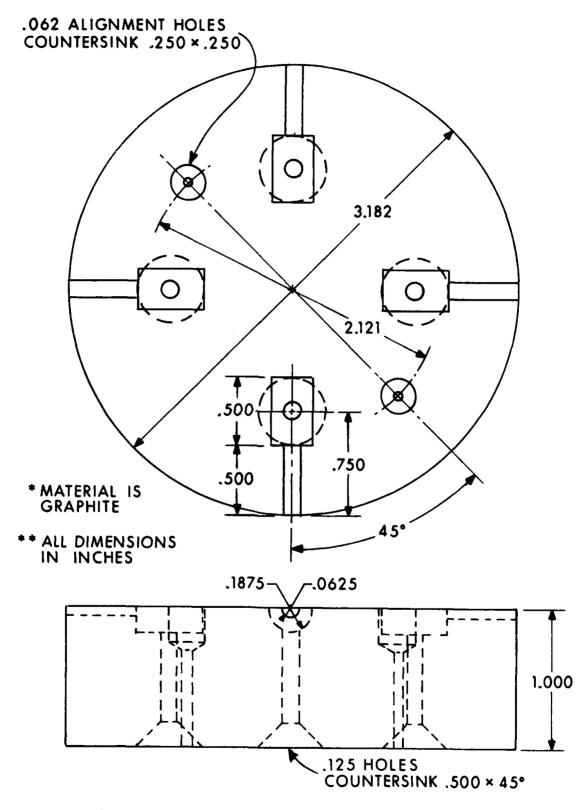
In addition, one batch of -325 mixed fines were retained for tests of the effects of mixed wide size range material. The composition of these mixed fines is 40% of -325 to +400, 50% of -400 to +500, and 10% of -500 mesh powder, all by volume. In this work, only two sintering temperatures were used, 650°C and 780°C. Times ranged from two hours to five hours. A completed sintered plug is shown in Figure 19. The tubing is standard 1/8 inch 0.D. copper, and is cut to a length of two inches and deburred. When placed in the mold, a caliper is used to insure correct centering of the tube end within the cylindrical cavity. Tubes are ultrasonically cleaned before being placed in the mold. The tubes are aligned four at a time in the mold bottom, and then the mold top placed on. Next, the desired powder is added to the cavities by pouring into the "funnels" formed in the mold, and tapping to distribute the powder and fill voids. The powder must be absolutely dry for this step. When no more powder can be added to the mold, the mold is placed in the reduction furnace and the sintering proceeds.

When completed, the plugs are removed from the sintering moulds and are installed in the housing shown in Figure 20. The completed assembly is shown in Figure 21. If operating temperatures in excess of 300°C are expected, the soft-soldered joints are replaced with silver soldered ones. The flow measurements were taken in the pressure range of 0 to 50 pounds/square inch, and will be given in these units since the commonly available pressure gauges are calibrated in these units. Flow rates will be given in liters/minute in an effort to adhere to S.I. units wherever practical. The calibration gas is



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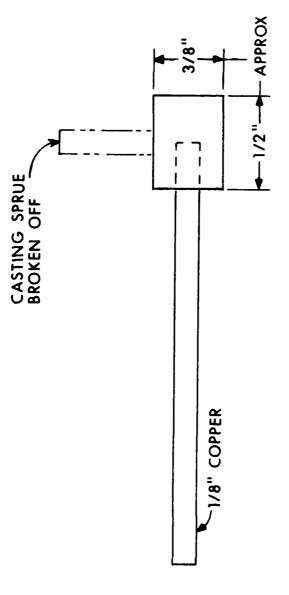
Figure 17. Flowmeter Plug Sintering Mould Bottom Walf



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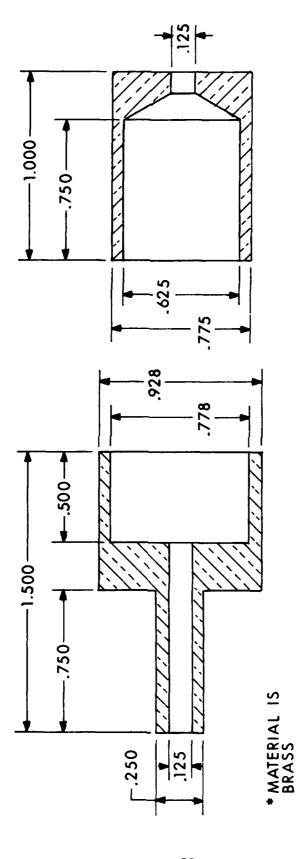
Figure 18. Flowmeter Plug Sintering Mould Top Ralf



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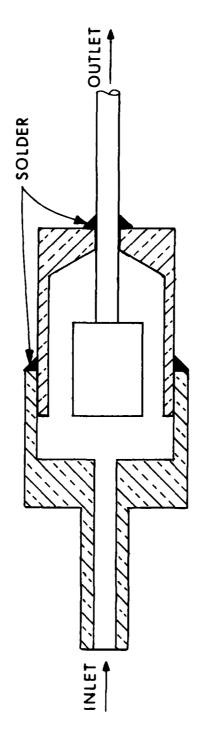
Figure 19. Dimensions of Completed Sintered Plug



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Figure 20. Flowmeter Housing Drawings

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Figure 21. Cutaway View of Assembled Sintered Plug Flowmeter

nitrogen, and a Precision Scientific Company wet test meter and stopwatch used to measure flows.

As this study was aimed at producing a usable set of flow constrictions, exhaustive tests on all possible meshes and times/temperatures were not measured in this work. The following is a table of a partial set of results from those combinations that yielded desirable and usable instruments:

Hesh size	Sintering Time	Degrees C	Flow at 5 PSI	Flow at 50 PSI	RMS from Linear	RMS from
-100 to +140	2.0 hours	650	1.386	7.067	2.34%	0.538%
-200 to +325	2.5 hours	650	0.372	2.941	3.31%	-
-325 mixed	2.0 hours	650	0.190	2.155	21 %	1.1%
-325 mixed	3.5 hours	780	0.109	1.351	13%	1.5%
-400 to +500	3.0 hours	650	0.634	4.248	2.1%	1.5%
-400 to +500	2.0 hours	780	0.645	1.289	28.2	1.2%
-400 to +500	3.5 hours	780	.0058	•0785	28.6%	1.7%

Figures 22 and 23 are least squares fits to actual measured flow vs. pressure for two of the plugs. Figure 22 represents a typical properly made plug, sintered of material with a low range of mesh sizes, with little evidence of melting on a microscopic scale. Figure 23 is data taken on a plug of mixed material, with evidence of slight melting during the sintering These representative sample runs, along with others, allow some qualitative conclusions to be drawn. Firstly, mixed fines with a large range of particle sizes is undesirable from the standpoint of linearity. This probably is due to the large range of pore sizes produced by the variation in particle size. Secondly, since great mechanical strength is not needed, lower sintering times are preferred for giving more nearly linear flow characteristics. Higher sintering temperatures cause more melting of the material, and consequently a wider range of pore sizes and poorer flow linearity. In the case of mixed fines, higher temperatures improve linearity probably due to the taking up of small particles into larger ones during the partial melting process. The main effect on porosity seems to be the particle size. The major effect on linearity seems to be the range of pore sizes, which is dependent on how close the sieve limits are, and what degree of melting occurs in the sintering process. Those plugs that are sintered at higher temperatures have more of a metallic shine than those done at lower temperatures. Under microscopic examination, there is more evidence of melting in these higher temperature ones.

Some additional notes from this work need to be mentioned. The plugs are excellent dampers of pulsations and oscillations in the gas flow. This has been used to great advantage in stabilizing the flame on the double knife edge burner previously referenced. An additional advantage of these flowmeters is that of resistance to plugging, and loss of calibration therefrom. For systems where the gases are to be delivered at normal atmospheric pressure, a measurement of the high side pressure is sufficient to establish reasonably accurate flows. For those applications where this is not the case, the scheme in Reference 7 should be used, substituting these plugs for the capillaries used therein. As a final note, manufacturers' names and model numbers used in

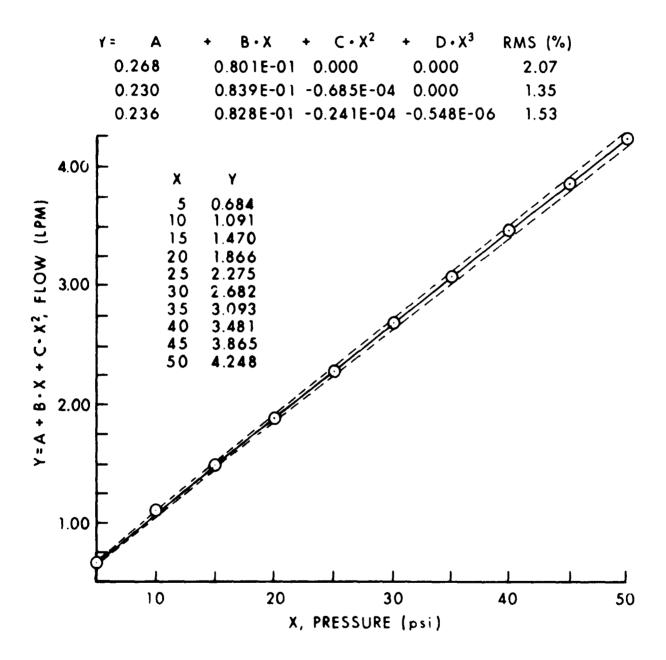
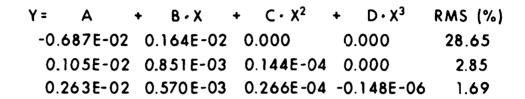


Figure 22. Calibration of Typical High Linearity Flowmeter



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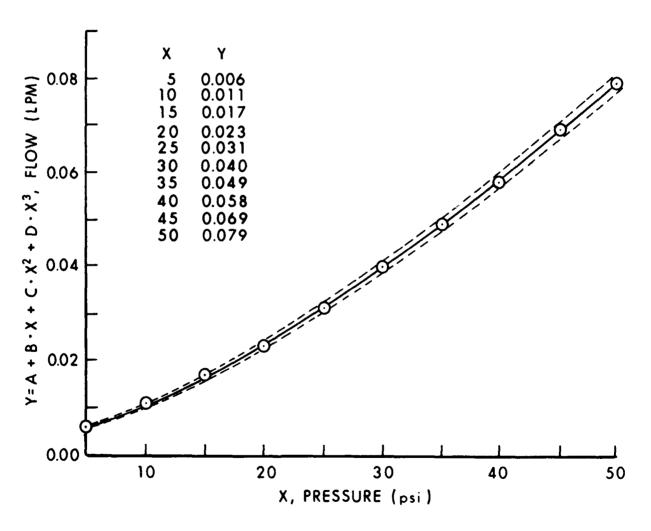


Figure 23. Calibration of Typical Low Linearity Flowmeter

this report are not to be construed to mean an endorsement by the U.S. government. They are included solely to provide a convenient means to reference additional specifications of those instruments and materials used. Any equivalent materials may be used.

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